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VELOCITY OF CRYSTALLIZATION OF SOLID PHASE COLLOIDS AT HIGH TEMPERATURES

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[Digest. Figures are appended.]

As distinguished from crystals occurring in solid metals, metal oxides, metal hydroxides, and other compounds often occur in the form of much smaller crystals having colloidal dimensions and consisting of a few scores or hundreds of atoms. This state, which differs qualitatively from both the crystalline and the amorphous-vitreous state, can be described as monophase colloidal.

While recrystallization of metals has been studied extensively, the process of crystal growth in monophase colloids has not been sufficiently investigated.

Typical examples of monophase colloids are the numerous varieties of "amorphous" coal, coke, carbon black, etc. When these materials are heated to temperatures of the order of 2,000-3,000° C, graphite, which is a genuine crystalline substance, is formed. In the course of the formation of graphite, the crystals grow from 10^{-7} to 10^{-3} cm and larger dimensions.

The rapid rate of growth of coal [carbon] crystals at high temperatures has been noted by many authors. These rates have not been measured, because the time required for recrystallization at high temperatures is of the order of seconds or a few minutes. It was difficult or impossible to carry out heating for such short periods in furnaces by means of the customary methods.

The following method was developed for investigating solid phase processes at high temperatures.

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The work was carried out in a furnace equipped with a carbon tube capable of being heated. This tube had a diameter of 35/45 mm and a length of 500 mm. Its contacts were water cooled. The carbon tube was placed in a casing supplied with a water jacket. The casing was filled with soot /carbon black/ which served as thermal insulation. At a constant network potential, the furnace reached a stationary state 1-1½ hours after the current had been switched on and the temperature remained constant ($\pm$ 20° C) for many hours.

The construction and mode of operation of this furnace are as described below. In the center of the vertically disposed carbon tube (Figure 1) a thick-walled graphite crucible is held by a carbon pin. The crucible has cylindrical walls and conically shaped openings both at the top and at the bottom. The opening at the bottom can be closed by means of a conical graphite stopper affixed to the end of a carbon shaft coming in through the bottom of the furnace. Using this appliance, the bottom opening of the crucible can be opened or closed at will. For measuring the temperature, a tube equipped with a parallel surface glass port is installed at the top of the furnace. The temperature of the crucible is measured through this glass port by means of an optical pyrometer. The tube equipped with the glass port also serves for the introduction of nitrogen and of the material to be investigated. The latter is introduced in a granular form through this tube from an attached test tube equipped with a rubber connection.

When the furnace has acquired a constant temperature, the test tube is raised, causing the grains of the material to drop into the crucible. Withdrawal of the crucible stopper after a definite time results in transfer of the material from the crucible to a receptacle attached to the bottom part of the furnace.

Material having grain dimensions of 0.5-1 mm is used for the experiments. The time necessary for establishing a constant temperature is calculated with a limit of error of 1% according to a formula proposed by the author (Doklady Akademii Nauk SSSR, Novaya Seriya, Vol LIX, 1105, 1948) and drawn up on the assumption that heat transfer proceeds by radiation only. The curve shown in Figure 2 depicts the relationship between the time necessary for temperature stabilization and the temperature of the furnace.

The quantity of material introduced simultaneously into the furnace was varied depending on the length of heating. With the shortest heating periods (5 seconds), 4-5 grains were used. With a 15-minute heating period, 50-70 grains were introduced simultaneously into the furnace, because in this case the duration of the temperature rise was not of great significance. This method of proceeding insured a temperature rise up to the temperature of the experiment rapid enough for its time to be neglected. The large mass of the graphite crucible guaranteed a practically constant temperature in it, the heat conductivity of graphite being high and the quantity of substance introduced into the crucible small.

Samples heated for 5 seconds, 30 seconds, and 1, 3, 5, and 15 minutes were prepared at the same time, and the heating of samples was alternated. For example, material to be heated for 5 seconds was introduced four times. It was also introduced four times when heated for 30 seconds, twice when heated for one minute, etc.

Thus, each treated sample represented an average of a great number of individual experiments. Alternation insured a good reproducibility and comparability of samples obtained at the same temperature, because the small and unavoidable fluctuations of furnace temperature were reflected in an equal manner on samples heated for different periods.

The constancy of the furnace temperature was checked every 15 minutes. Usually the temperature stayed at a constant level. In cases when the temperature did fluctuate considerably (over more than 30°) due to a changing resistance, an old tube, or some other accidental condition, the furnace could not be brought back precisely to the original temperature. Unless a sufficient number of samples had been accumulated up to that time, the whole series of experiments was considered faulty and its results were discarded.

The growth of crystals of all types of carbonaceous material is accompanied by a diminution of the electrical resistance. This permitted conductivity to be used as an index of comparative crystal size.

For conductivity measurements, the samples were powdered and sifted through a sieve having 1,600 holes per sq cm. The conductivity of the sifted powder was determined at a pressure of 170 kg per sq cm by means of a balanced circuit method eliminating the effect of the resistance arising at the contact between the powder and the compressing piston.

For each sample, the resistance was measured 5-7 times and the average value taken. The maximum deviation from the mean comprised about 4%. The results of these measurements are shown in the curves of Figure 3, which represent the dependence of the rate of crystal growth on time at various temperatures.

Petroleum coke which had been previously treated at 1,200° C was investigated.

At the same time (by heating it for 1 minute and 15 minutes at a temperature of about 2,500° C) petroleum coke which had been purified in the laboratory was investigated, so that the effect of small quantities of ash constituents contained in the initial material could be studied. Purification was carried out by repeatedly boiling out the material in hydrochloric acid (1:3) and then heating it for 2 hours at 900° C in a current of chlorine. This was followed by another washing with hydrochloric acid and then washing with boiling water. Material treated in this manner contained only traces of /inorganic/ admixtures. No difference in the behavior of samples thus treated could be established.

As can be seen from the curves, the process of crystallization is extremely rapid. The rate of crystal growth rapidly increases with increasing temperatures.

It is probable that with longer periods of heating of the carbons obtained by the above-described method, the growth of crystals will continue. However, the growth rate at high temperatures becomes so small at the expiration of 5-15 minutes that one may speak of a quasi-equilibrium characterized by a certain mean crystal size. This size depends on the temperature of the preceding treatment and the properties of the initial material.

/Appended figures follow/

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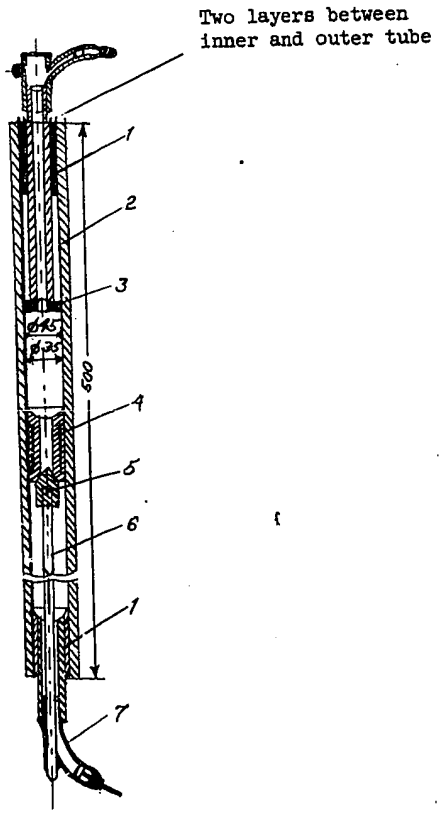

Figure 1. Apparatus for Carrying Out Treatment of Samples for Short Periods at High Temperatures (2,000-3,000° C).

(1) porcelain tube, (2) furnace tube, (3) disk, (4) crucible, (5) conical stopper, (6) carbon shaft, (7) copper receptacle.

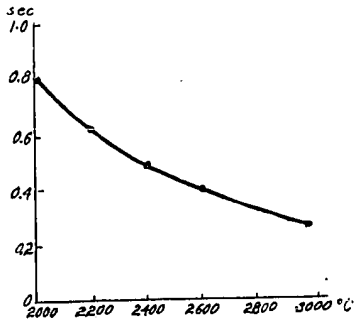

Figure 2. Time Necessary for Equalizing the Temperature (With a Precision of 1%) Between the Furnace and a Carbon Sphere Having a Diameter of 0.1 Cm, When Heat Transfer Takes Place by Means of Radiation Only.

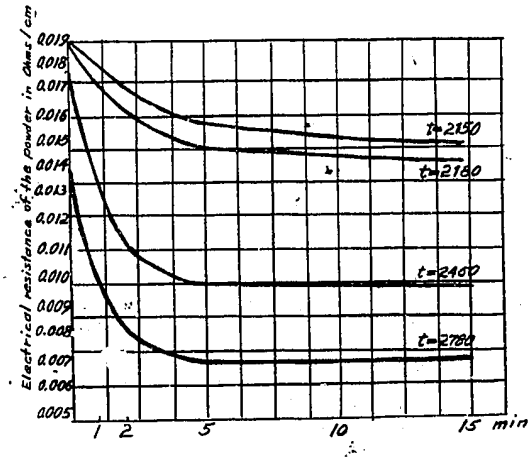

Figure 3. Dependence of the Electrical Resistance of Powder Being Graphitized on the Time of Heating and the Temperature.

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